Table 2. *Selected intramolecular distances (Å) and angles (°) with estimated standard deviations in parentheses*

Ir—N(1)	2·103 (8)	Ir—N(2)	2·084 (8)
Ir—C(1)	2·131 (12)	Ir—C(2)	2·129 (11)
Ir—C(5)	2·091 (11)	Ir—C(6)	2·127 (11)
Ir—M1	2·005	Ir—M2	1·997

M1—Ir—M2	88·7	M1—Ir—N(1)	93·2
M1—Ir—N(2)	175·8	M2—Ir—N(1)	178·1
M2—Ir—N(2)	93·7	N(1)—Ir—N(2)	84·5 (3)

M1 and M2 are the midpoints of the C(1)—C(2) and C(5)—C(6) bonds.

Related literature. This complex is the immediate precursor of [Ir(cod)(py)(PCy₃)], which has been of considerable value as a hydrogenation catalyst (Crabtree, 1979). The conformations of cod in [{Ir-(cod)Cl}₂] and [Ir₂(cod)₂Cl₂(μ₂-OH)₂(μ-O)] (Cotton, Lahuerta, Sanau & Schwotzer, 1986) are similar to those observed here. The iridium nitrogen bonds are comparable in length with that found in [Ir(cod)-(py)(PCy₃)][PF₆], with the bonds to the diene being slightly shorter, probably reflecting the lower degree of steric crowding (Abbassioun, Hitchcock & Chaloner, 1989). There are many examples of structures of [L_2Rh(diene)]⁺ complexes (Knowles, 1983; Anderson & Pignolet, 1981); the geometries about the metal are all similar.

We thank Johnson Matthey for the loan of iridium complexes.
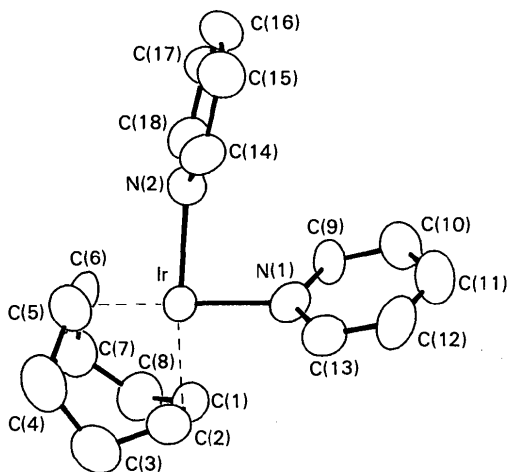

Fig. 1. Molecular structure and numbering scheme for [Ir(py)₂(cod)]⁺.

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Structure of Diaqua(benzoato)chlorodioxouranium(VI)

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Abstract. [U(C₇H₅O₂)ClO₂(H₂O)₂], $M_r = 462·63$, orthorhombic, *Pnna*, $a = 8·0247(15)$, $b = 9·7759(9)$, $c = 13·7205 (12)$ Å, $V = 1076·3 (5)$ Å³, $Z = 4$, $D_x = 2·855 (1)$ g cm⁻³, $\lambda(Mo K\bar{\alpha}) = 0·71073$ Å, $\mu = 145·56$ cm⁻¹, $F(000) = 832$, $T = 297$ K, $R = 0·024$ for 533 observations with $I > 3\sigma(I)$. The structure is of molecular type: the individual molecules are bisected by the diad axis parallel to the [001] direction, on which lie U, Cl and three C atoms. The water molecules are engaged in intermolecular hydrogen bonding with Cl atoms [O(3)–Cl = 3·15 (1) Å] and carboxylic O atoms [O(3)–O(2) = 2·70 (1) Å].

Experimental. Yellow single crystal of dimensions 0·75 × 0·100 × 0·150 mm, grown during the reaction between UO₂Cl₂ and C₆H₅COOAg in aqueous solution, Enraf–Nonius CAD-4 diffractometer with graphite monochromator, lattice parameters by least-squares procedure from setting angles of 25 reflections, θ between 8 and 12°, collection of data up to $2\theta = 50°$, measured for $0 \le h \le 9$, $0 \le k \le 11$, $0 \le l \le 16$ using an $\omega/2\theta$ scan technique, corrections for background and Lorentz–polarization effects, negligible loss of intensity from three standard reflexions remeasured every hour, empirical absorption correction based on ψ scans of top reflexions applied, max. and min. transmissions 0·896 and 0·997. 1155 reflexions measured, 947 unique. Structure solved by Patterson method, using the *Enraf–Nonius SDP* programs (Frenz, 1983); H atoms of the phenyl ring introduced with ideal

Table 1. *Positional and thermal parameters*

$$B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i\cdot\mathbf{a}_j.$$

	x	y	z	B_{eq} (Å^2)
U	0·250	0·00	0·16926 (2)	1·616 (9)*
Cl	0·250	0·00	−0·0293 (3)	2·71 (8)*
O(1)	0·403 (1)	0·1302 (9)	0·1685 (7)	2·4 (2)
O(2)	0·339 (1)	−0·0853 (9)	0·3303 (7)	2·3 (2)
O(3)	0·472 (1)	−0·164 (1)	0·1407 (7)	2·5 (2)
C(1)	0·250	0·00	0·381 (1)	2·3 (3)
C(2)	0·250	0·00	0·485 (1)	2·7 (3)
C(3)	0·316 (2)	−0·110 (2)	0·540 (1)	3·3 (3)
C(4)	0·315 (2)	−0·111 (2)	0·637 (1)	3·0 (3)
C(5)	0·250	0·00	0·687 (1)	3·7 (4)
H(3)	0·363	−0·185	0·506	5·0
H(4)	0·355	−0·189	0·671	5·0
H(5)	0·250	0·00	0·756	5·0

* These atoms were refined anisotropically.

Table 2. *Selected interatomic distances* (Å) *and angles* (°)

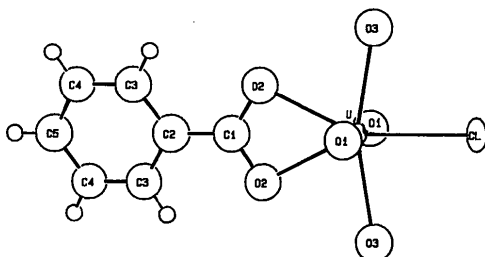|			
U–Cl	2·725 (4)	C(1)–O(2)	1·31 (1)
U–O(1)	1·767 (9)	C(1)–C(2)	1·43 (2)
U–O(2)	2·467 (9)	C(2)–C(3)	1·42 (2)
U–O(3)	2·429 (9)	C(3)–C(4)	1·32 (1)
		C(4)–C(5)	1·39 (2)
Cl–U–O(1)	89·7 (4)	O(2)–C(1)–O(2^i)	115 (1)
Cl–U–O(2)	153·6 (3)	C(1)–C(2)–C(3)	122·7 (7)
Cl–U–O(3)	80·7 (2)	C(3)–C(2)–C(3^i)	115 (1)
O(1)–U–O(2)	92·8 (4)	C(2)–C(3)–C(4)	123 (1)
O(1)–U–O(3)	88·1 (4)	C(3)–C(4)–C(5)	120 (2)
O(2)–U–O(3)	73·1 (3)	C(4)–C(5)–C(4^i)	120 (1)
O(1)–U–O(1^i)	179·3 (3)		
O(2)–U–O(2^i)	52·8 (2)		
O(3)–U–O(3^i)	161·4 (2)		

Symmetry code: (i) $\frac{1}{2}-x, \bar{y}, z$.
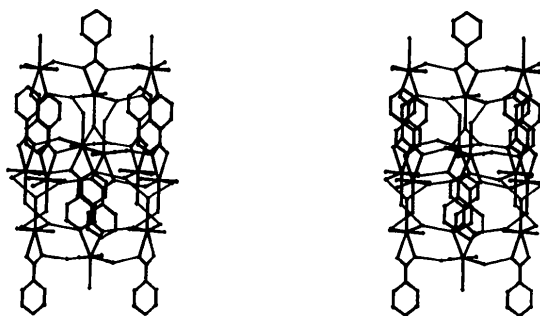

Fig. 1. *ORTEP* drawing of the molecule.

Fig. 2. Stereoview of the structure, with hydrogen bonding.

geometry (C—H 0·95 Å) with thermal parameters fixed at 5 Å^2, but not refined; complex atomic scattering factors from *International Tables for X-ray crystallography* (1974). Only U and Cl atoms were given anisotropic thermal parameters. Full-matrix least-squares refinement on F. Final R values: R_F 0·024, $R_{F^2}(w)$ 0·030 (unit weights). $(\Delta/\sigma)_{max} = 0·21$. Max. height in final difference Fourier synthesis 0·84 e Å^{-3}. The atomic parameters are given in Table 1,* selected interatomic distances and angles in Table 2.

The U atom, as the uranyl ion, has its classical environment: a pentagonal bipyramid, with the quasi-linear axis O(1)—U—O(1′) 179·3 (3)°, approximately perpendicular to the pentagonal base. The deviations from the mean plane between U and its neighbours in the irregular pentagon made of two O atoms from a bidentate carboxyl group, two O atoms from two water molecules and one Cl atom, are the following: U and Cl 0, two O(3) ±0·05 (1), two O(2) ±0·12 (1) Å. The intra-non-bonding contacts in the pentagon are: Cl—O(3) 3·34 (1), O(2)—O(2′) 2·20 (1) and O(2)—O(3) 2·91 (1) Å.

Fig. 1 (Johnson, 1976) gives the molecular structure with atom numbering. The most interesting feature is the hydrogen-bonding scheme, which ensures the cohesion of the structure (Fig. 2). Each O(3) atom (H$_2$O molecule) is involved in intermolecular bonds with a carboxyl atom O(2) and a Cl atom, with bond lengths respectively 2·70 (1) and 3·15 (1) Å, and angle Cl—O(3)—O(2) 144·5 (4)°. These values are close to those reported in the literature, basically for O—Cl bonds: 3·18 Å in CuCl$_2$.2H$_2$O (Peterson & Levy, 1957) with angle O—H—Cl 164°; they also suggest the non-linearity of the hydrogen bonds. Each Cl atom is hydrogen bonded to two O(3) atoms which belong to two different H$_2$O molecules packed along the x axis: this leads to a strong cohesion of layers parallel to the (010) plane. The geometry around the Cl atom, including the U atom, is almost planar with two Cl—O(3) distances of 3·15 Å, one Cl—U distance of 2·724 (4) Å and angles O(3)—Cl—O(3) 121·9 (3) and U—Cl—O(3) 119·1 (2)°.

The intermolecular hydrogen bonds between O(3)—O(2) are then responsible for the cohesion in the third direction, y.

The phenyl groups appear in the packing in two orientations at ±26° from the (100) plane.

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Structure of (R*,R*)-3-Methyl-1-(4-methyl-5-oxo-2-pyrrolidinyl)-3-pyrrolin-2-one

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Abstract. $C_{10}H_{14}N_2O_2$, $M_r = 194.23$, orthorhombic, $P2_12_12_1$, $a = 5.977$ (5), $b = 10.067$ (8), $c = 17.406$ (14) Å, $V = 1047.4$ (1.5) Å^3, $Z = 4$, $D_m = 1.2$ (1), $D_x = 1.23$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 0.676$ mm^{-1}, $F(000) = 416$, room temperature, $R = 0.053$ for 725 observed reflections. The unsaturated lactam ring is planar, the saturated ring has an envelope conformation with C(24) out of plane. Ring substituents can be described as O(27) equatorial, C(26) pseudo-equatorial and N(11) pseudo-axial. Only the relative configurations of the chiral centres were established. There is an intermolecular hydrogen bond N(21)–H(22)···O(27^i) [(i) $x-0.5$, $0.5-y$, $-z$] with N···O = 2.916 (5), H···O = 1.91 Å, N–H···O = 154°.

Experimental. The title compound was isolated from Lilium candidum L. (Liliaciae) by Haladová, Eisenreichová, Bučková, Tomko & Uhrin (1988). Colourless crystal $0.6 \times 0.1 \times 0.07$ mm, D_m by flotation. Space group from Weissenberg photographs. Syntex $P2_1$ diffractometer, graphite monochromator, lattice parameters from 15 reflections $22 < 2\theta < 50°$, $\omega-2\theta$ technique, scan rate $4.88-29.3°$ 2θ min^{-1}, scan range $2.0°$ plus $K\alpha_1, K\alpha_2$ difference, background at each end of the scan for half of the scan time, two standards (014, 122) monitored every 98 reflections did not vary significantly, 902 reflections measured for $2\theta < 120°$ ($h = 0$ to 6, $k = 0$ to 10, $l = 0$ to 18), $(\sin\theta/\lambda)_{max} = 0.546$ Å^{-1}, 844 unique, 119 unobserved with $I < 1.96\sigma(I)$. Lorentz and polarization but no absorption or extinction corrections. Structure solved by MULTAN80 (Main et al., 1980), refinement on $|F_o|$ by

Table 1. Final atomic coordinates ($\times 10^4$) with e.s.d.'s in parentheses and equivalent isotropic thermal parameters

$$B_{eq} = \tfrac{4}{3}\Sigma_i \beta_{ij} a_i^2.$$

	x	y	z	B_{eq}(Å^2)
N(11)	7680 (6)	5082 (4)	1442 (2)	4.94
C(12)	6172 (7)	6069 (5)	1572 (3)	5.24
C(13)	7034 (8)	7281 (4)	1189 (3)	5.49
C(14)	8909 (9)	6969 (5)	842 (3)	6.15
C(15)	9541 (8)	5541 (5)	968 (3)	5.64
C(16)	5763 (11)	8560 (6)	1243 (3)	7.73
O(17)	4429 (6)	5936 (3)	1935 (2)	6.91
N(21)	6734 (6)	2922 (4)	956 (2)	4.79
C(22)	8345 (7)	2097 (4)	724 (2)	4.19
C(23)	10289 (7)	2188 (4)	1282 (3)	4.56
C(24)	9317 (8)	2957 (5)	1966 (3)	5.56
C(25)	7259 (7)	3692 (5)	1647 (3)	4.85
C(26)	11286 (10)	853 (5)	1470 (4)	7.82
O(27)	8258 (5)	1390 (3)	151 (2)	4.99

Table 2. Interatomic distances (Å) and angles (°) with e.s.d.'s in parentheses

N(11)–C(12)	1.361 (6)	C(21)–C(22)	1.333 (5)
N(11)–C(15)	1.461 (6)	N(21)–C(25)	1.466 (6)
N(11)–C(25)	1.466 (6)	C(22)–C(23)	1.517 (6)
C(12)–C(13)	1.483 (7)	C(22)–O(27)	1.227 (5)
C(12)–O(17)	1.226 (6)	C(23)–C(24)	1.534 (6)
C(13)–C(14)	1.311 (7)	C(23)–C(26)	1.505 (7)
C(13)–C(16)	1.498 (8)	C(24)–C(25)	1.539 (7)
C(14)–C(15)	1.502 (7)		

C(12)–N(11)–C(15)	111.5 (4)	C(22)–N(21)–C(25)	115.0 (4)
C(12)–N(11)–C(25)	122.9 (4)	N(21)–C(22)–C(23)	108.8 (4)
C(15)–N(11)–C(25)	124.8 (4)	N(21)–C(22)–O(27)	125.2 (4)
N(11)–C(12)–O(17)	124.7 (4)	C(23)–C(22)–O(27)	126.0 (4)
N(11)–C(12)–C(13)	107.2 (4)	C(22)–C(23)–C(24)	103.7 (4)
C(13)–C(12)–O(17)	128.1 (4)	C(24)–C(23)–C(26)	115.6 (4)
C(12)–C(13)–C(14)	107.9 (4)	C(22)–C(23)–C(26)	112.9 (4)
C(12)–C(13)–C(16)	120.2 (4)	C(23)–C(24)–C(25)	105.4 (4)
C(14)–C(13)–C(16)	132.0 (5)	N(11)–C(25)–N(21)	110.0 (4)
C(13)–C(14)–C(15)	112.2 (5)	N(11)–C(25)–C(24)	114.2 (4)
N(11)–C(15)–C(14)	101.2 (4)	N(21)–C(25)–C(24)	102.3 (4)

N(11)–C(12)–C(13)–C(14)	1.7 (5)	C(13)–C(14)–C(15)–N(11)	0.6 (6)
N(11)–C(12)–C(13)–C(16)	−178.6 (4)	C(13)–C(12)–N(11)–C(25)	−171.8 (4)
N(21)–C(22)–C(23)–C(24)	12.4 (4)	C(14)–C(15)–N(11)–C(12)	0.6 (5)
N(21)–C(22)–C(23)–C(26)	138.2 (4)	C(14)–C(15)–N(11)–C(25)	170.8 (4)
O(17)–C(12)–C(13)–C(14)	−177.5 (5)	C(15)–N(11)–C(12)–C(13)	−1.4 (5)
O(17)–C(12)–C(13)–C(16)	2.2 (8)	C(15)–N(11)–C(25)–C(24)	49.5 (6)
O(17)–C(12)–N(11)–C(25)	7.5 (7)	C(15)–N(11)–C(25)–N(21)	−64.8 (5)
O(27)–C(22)–C(23)–C(24)	−167.9 (4)	C(16)–C(13)–C(14)–C(15)	178.9 (5)
O(27)–C(22)–C(23)–C(26)	−42.1 (6)	C(22)–C(23)–C(24)–C(25)	−20.6 (4)
O(27)–C(22)–N(21)–C(25)	−178.2 (4)	C(23)–C(24)–C(25)–N(21)	21.0 (4)
C(12)–C(13)–C(14)–C(15)	−1.4 (6)	C(26)–C(23)–C(24)–C(25)	−144.6 (4)
C(12)–N(11)–C(25)–C(24)	−141.4 (4)	C(24)–C(25)–N(21)–C(22)	−14.5 (5)
C(12)–N(11)–C(25)–N(21)	104.3 (5)	C(25)–N(21)–C(22)–C(23)	1.5 (5)
		O(17)–C(12)–N(11)–C(15)	177.9 (4)